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SPECIAL EQIPMENT FOR ETCHING NITROCELLULOSE FILM

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Risø National Laboratory, DK-4000 Roskilde, Denmark
August 1983

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SPECIAL EQUIPMENT FOR ETCHING
NITROCELLULOSE FILM

by
J.C. Domanus

Nuclear Department
Elsinore Shipbuilding and Engineering Co., Ltd.*

1. INTRODUCTION

Unlike the case of X-ray film, where processing equipment is commercially available, no such equipment exists for processing nitrocellulose film used in neutron radiography. Thereafter, after being exposed to neutrons, this film must be etched at constant temperature to make the radiographic image produced on it visible during neutron radiography.

There is also no commercially available equipment for viewing processed neutron radiographs on nitrocellulose film.

This deplorable situation is caused by the limited use of nitrocellulose film for neutron radiography compared with industrial X-ray film. Therefore, neither manufacturers of nitrocellulose film nor X-ray film accessories are interested in putting equipment on the market for processing and viewing the nitrocellulose film used for neutron radiography.

In this situation most of the users of nitrodellulose film for neutron radiography process the film in rather primitive equipment, which they are obliged to produce themselves. (Even the manufacturer of the nitrocellulose film has no special equipment for its processing.)

---

* Work performed under contract with Risø National Laboratory

To alleviate those difficulties Risø National Laboratory has decided to design, construct, and produce special equipment for etching nitrocellulose film used at Risø for neutron radiography.

The design was based on three requirements:

- The size of the processed film will be 9 x 12 cm (this being the standard size of Kodak-Pathé nitrocellulose film).
- Temperature of the etching bath must be kept constant (but can be chosen over a wide range).
- The etching bath should not be agitated during etching (as required by the nitrocellulose film manufacturer.)

The other problem connected with the application of nitrocellulose film to neutron radiography - the viewing of the neutron radiographs - has not been solved yet.

2. NITROCELLULOSE FILM FOR NEUTRON RADIOGRAPHY

In practice, there is only one nitrocellulose film available on the market for neutron radiography. It is manufactured by Kodak-Pathé. The previous CA 80-15 film (rose tinted) was recently superseeded by the CN 85 film (colourless).

Both films are described by Kodak in the following way:

> "KODAK CA 80-15 and CN 85 film consists of 100 μm thick sheet of cellulose nitrate. Film Type B and Type 1 B are two variations of the above film, being coated with lithium borate dispersed in a water-soluble binder which acts as a converter screen by means of the (n,α) reaction. This screen is coated on both surfaces of the Type B Film, but only on one surface of the Type 1 B Film.
>
> Using these films it is possible to obtain:
>
> • a neutron radiographic image by means of the (n,α) reaction in the screen,
>
> • an autoradiograph of any substance which emits alpha rays naturally, or of any object containing elements such as boron or lithium which become alpha emitters when they are irradiated with neutrons.

The use of a film incorporating its own converter screen is recommended because of more uniform contact between the film and the converter screen which greatly reduces the deleterious effect of dust specks. It is for this reason that the Type B and Type 1 B Films have been developed."

Their application to neutron radiography is described as follows:

"The use of these films not only eliminates the prolonged and critical handling of activated screens - previously required by classical methods - but also enables much higher resolving power to be achieved. In addition, the insensitivity of these films to gamma rays allows unfiltered neutron fluxes to be used. Furthermore, this absence of gamma-ray sensitivity enables neutron radiographs to be made of very active samples such as irradiated fuel elements.

KODAK CA 80-15 and CN 85 Films are also suitable for neutron radiography with fast neutrons, especially in medical and biological applications."

About the processing and evaluation, KODAK gives the following recommendations:

"The tracks detected by these films are not directly visible but have to be intensified by processing the films in an alkaline bath.

- The recommended etching agent is a 10% (2.5 N) solution of caustic soda (sodium hydroxide), of analytical purity, in distilled water.

- After this process the film should be washed in water for at least 30 minutes.

- The strength of the bath affects the results obtained and it must therefore be prepared exactly and maintained at the correct strength. Care must be taken, particularly when processing at 60°C, to allow for the effect of evaporation and that of carbonation (the absorption of atmospheric carbon dioxide which combines with the caustic soda to form less-alkaline sodium carbonate).

- However, it is not recommended to exceed a concentration of 6 N (24%), in this case it would be necessary to wash with plenty of water and continuous agitation (25 to 30°C).

- Etching may be carried out at either 60°C or 25°C.

- The etching bath should not be agitated. A uniform temperature should be maintained either by using a water or jacket or, more satisfactorily, by means of a thermostatically controlled environment, such as a hot-air cabinet.

- Surface-active agents (such as detergents), organic solvents, adhesive tapes, and ballpoint or felt-pen inks may lead to adverse reactions and therefore should not be allowed to contaminate the etching bath.

In specific applications the parameters associated with the process may be varied to obtain optimum results.

The etching time may be varied as below in accordance with the degree of irradiation:

From 10 minutes to 30 minutes at 60°C.
From 2 hours to 8 hours at 25°C.

A low level of irradiation followed by longer etching will result in increased sensitivity and higher contrast. On the other hand, a higher level of irradiation followed by shorter etching time will result in a lower-contrast image, but with finer definition.

When a low-intensity source is used, the KODAK converter screen BN 1 can be an appropriate help.

The examination of the image originally obtained will be facilitated if an enlargement of it is made on a high-contrast photographic printing paper or film such as KODABROM II High-Contrast RC Paper or Bromide Paper or a KODALITH graphic-arts contact Film.

KODAK CA 80-15 and CN 85 Films, Type B and Type 1B must be washed in water after irradiation, but before etching, to remove the converter screen. They may then be regarded as equivalent to KODAK CA 80-15 and CN 85 Films.

Finally, these films should be stored before use in a cool, dry place (4°C with maximum relative humidity of 50%). Ensure that they have reached ambient temperature before using them."

About the availability of those films the following can be quoted from Kodak pamphlets:

"• Stock items: KODAK CA 80-15 Film
Catalogue N° 0984
Box of 25 sheets, format 9 x 12 cm.

• Special Order items:
Minimum order: 3 m²; maximum roll width: 1.10 m
Length between 6 and 100 m.
Delivery time: about 3 months.

• Experimental items available on Special Order:
Applicable only to KODAK CA 80-15 Films,
Type B and Type 1B.
Minimum order: 1 m².
Delivery time: about 3 months.
KODAK CA 80-15 Films, Type B and Type 1B:
maximum width 200 mm, maximum length 10 m.

• Stock items: KODAK CN 85 Film
Catalogue N° 503 3350
Box of 25 sheets, format 9 x 12 cm

- Special Order items:
  Minimum order: 3 m^2; maximum roll width: 1.10 m;
  Length between 6 and 100 m.
  Delivery time: about 3 months.

- Other items available on Special Order:
  Applicable only to KODAK CN 85 Films, Type B and Type 1 B.
  Minimum order: 1 m^2.
  Delivery time: about 3 months.
  Size: maximum width 250 mm, maximum length 5 m."

3. CONVERTER SCREENS

As described in 2 above the Kodak CA 80-15 and CN 85 films, type B and type B 1 are coated on both or on one side by a converter screen.

They can, however, be used without this coating (a simple CA 80-15 and CN 85 films). Then a Kodak Converter Screen BN 1 ought to be used with those films for neutron radiography.

The BN 1 screen is described by Kodak in the following way:

> "KODAK Converter Screen BN 1 is made from natural boron and is a neutron-alpha converter designed for thermal and epithermal neutron radiography as well as for dosimetry and detection of these neutrons.
>
> It should normally be used with KODAK CN 85 and LR 115 type 1 and type 2 films.
>
> **Features:**
> This converter is coated on a 100-micron thick very stable polyester base.
>
> Under the action of thermal and epithermal neutrons the converter screen BN 1 responds according to the well-known reaction:

$$ {}^{10}_{5}B + {}^{1}_{0}n \longrightarrow {}^{7}_{3}Li + {}^{4}_{2}He(\alpha) $$

The efficiency of KODAK BN 1 is particularly high (about 2.5 times higher than that of the lithium borate coated on KODAK CN 85 type 1 B film).

Instructions for use:

Before use, check that the surface of KODAK Converter Screen BN 1 is free of dust particles.

If this is not the case put it in an ionization chamber for a while. Also, check that mat side of the screen is toward the sensitive layer of the film.
When handling the converter screen, avoid finger marks on the boron as well as abrasions from chemical and/or mechanical agents.

These precautions allow indefinite reuse of KODAK Converter Screen BN 1.

It is at last strongly recommended to establish perfect contact between the converter screen and the cellulose nitrate layer of the film. This is quite possible with a vacuum cassette.

Availability:

KODAK Converter Screen BN 1 is sold in the following sizes:

- Catalogue number 5033592 box of 10 screens 8 x 20 cm
- Catalogue number 5033600 box of 5 screens 20 x 25 cm

Minimum order quantity in each size is one box and delivery time is about 2 to 3 months.
For other sizes, please apply to KODAK in your country."

4. VISUALIZATION OF NEUTRON RADIOGRAPHS

The radiographic image on a nitrocellulose film is not so easy to assess as that on an X-ray film. Therefore it was already mentioned above in 2 that the examination of the radiographic image can be facilitated by copying it from the nitrocellulose film on high contrast paper or film.

There is, however, another method, recommended by KODAK, for directly viewing neutron radiographs on nitrocellulose film. Kodak describes it as follows:

> "When the cellulose nitrate film is used for non-destructive testing, the image obtained shall be as legible as possible; in this respect, a projection print is commonly made on a high-contrast photographic paper or film. This technique is rather long, since, after development of the original print, it necessitates the formation of a photographic copying negative by projection. Moreover, most users do not have large size enlargers, so that use is practically limited to a 9 x 12 cm size, for example.
>
> It is now proposed to obtain the direct visual examination of track-etched neutron radiographs exactly as for silver conventional X-ray films as a transparency through a negatoscope.
>
> The etched tracks obtained on the cellulose nitrate film form a translucent area and the density of difference between translucent and non-translucent areas is weak: the use of a negatoscope is therefore not possible.
>
> According to the disclosed method, the lack of density difference is overcome by the use of polarizing filters: the neutron radiograph to be examined is sandwiched between two polarizers, one of these polarizers being rotated 90° from the position of the other in order to obtain complete light extinction. The sandwich is exposed to a diffused light.

As a result, materials having a weak neutron absorption correspond on the cellulose nitrate film to looking translucent areas; these areas are converted by polarizers into high-light areas. Conversely, the materials having a strong neutron absorption correspond to high-density areas. The disclosed method permits us to view large size neutron radiographs of any form whatever, e.g., either as plates or webs or stripes.

It is also possible to photograph the image modified by the polarizers or to measure the optical densities with a densitometer, whereby quantitative measurements are obtained."

This method was tested at Risø using Polaroid circular polarizers HNCP 37 Neutral.

5. DESIGN CRITERIA FOR ETCHING EQUIPMENT

As mentioned already in 1 above the design of the Risø special equipment for etching nitrocellulose film is based on: the size of the films to be etched, the temperature of the etching bath, and the non-agitation condition of the bath. From these data, the following design criteria can be deduced:

1) To facilitate handling of the 9 x 12 cm films standard X-ray film hangers ought to be used. Such film hangers are commercially available. They can accommodate one, two, or three 9 x 12 cm films on one hanger, as shown in fig. 1.

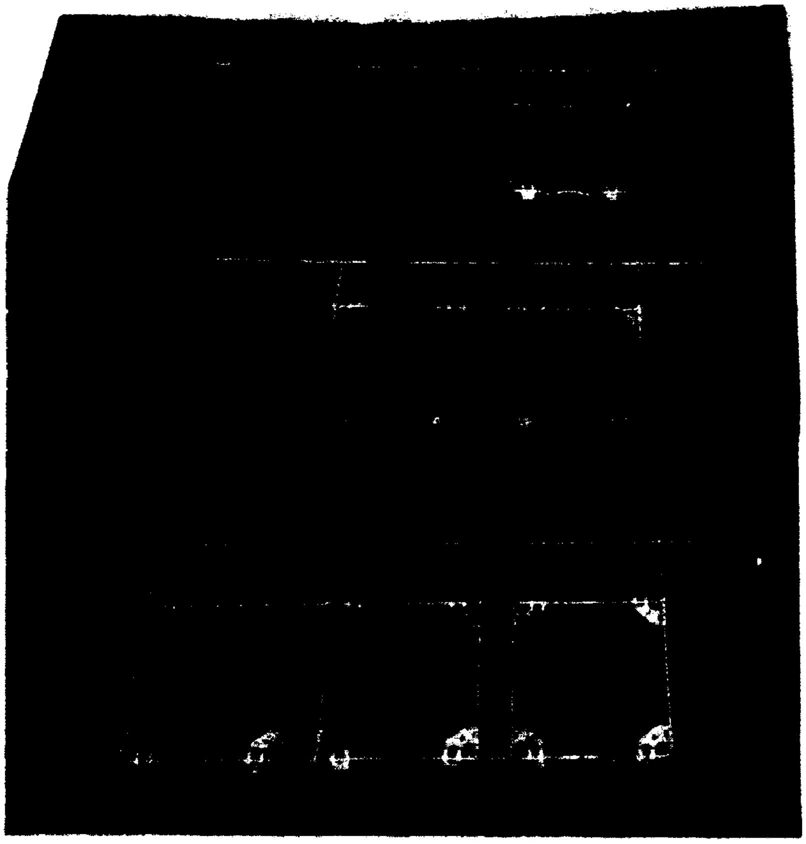

Fig. 1. Single, double, and triple film hangers

2) To be able to etch several 9 x 12 cm films simultaneously, the etching tank was designed to accommodate up to 5 film hangers. Thus, the maximum capacity of the etching tank is 15 films of 9 x 12 cm (see fig. 2.)

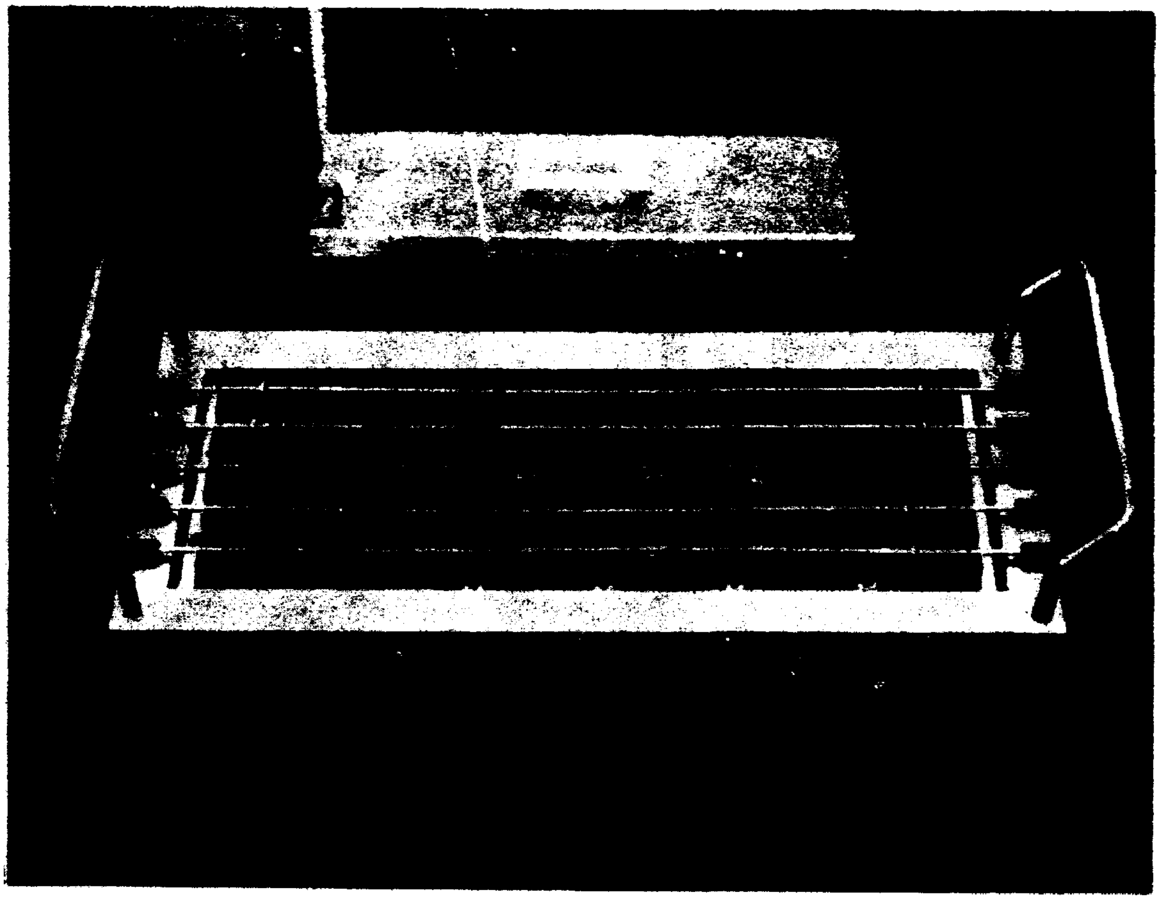

Fig. 2. Etching tank accommodating up to 5 film hangers

If films of other sizes are to be etched, the tank provides also for this possibility. Fig. 3 shows, e.g. a film hanger for 10 x 24 cm films. Five such film hangers can be placed in the etching tank simultaneously.

Fig. 3. 10 x 24 cm film hanger

The films on hangers are evenly spaced in the tank by special notches. Thus, the films will not touch each other during etching.

3) The etching tank itself is made of stainless steel. A glass thermometer can be inserted through to control the temperature of the etching bath (see fig. 4.)

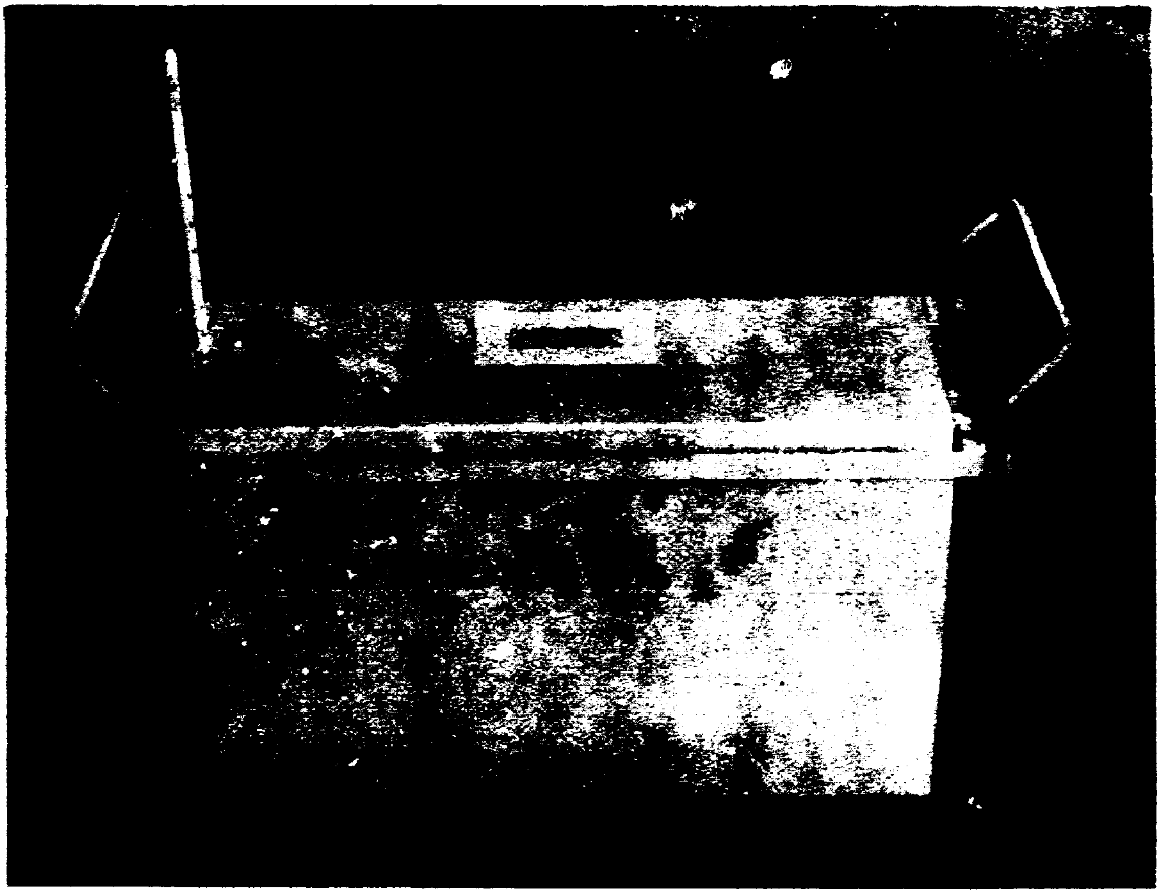

Fig. 4. Stainless steel etching tank with
stainless steel lid (and thermometer)

4) As mentioned above in the description of the nitrocellulose films, they can be etched at various etching bath temperatures and whatever temperature is chosen ought to be kept constant during etching. For that purpose a Heto Hetotherm temperature control system was used. It is shown in figs. 5 and 6.

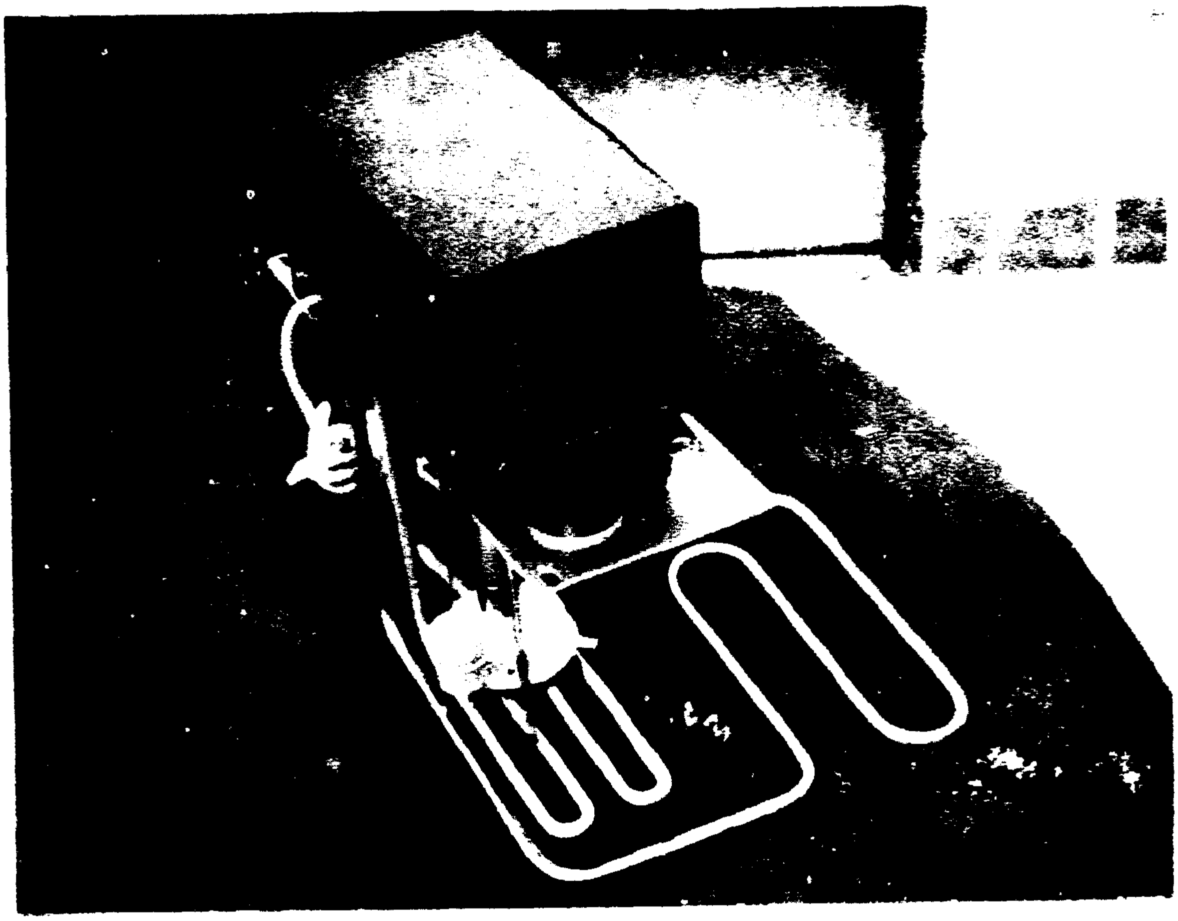

Fig. 5. Hetotherm temperature control system.

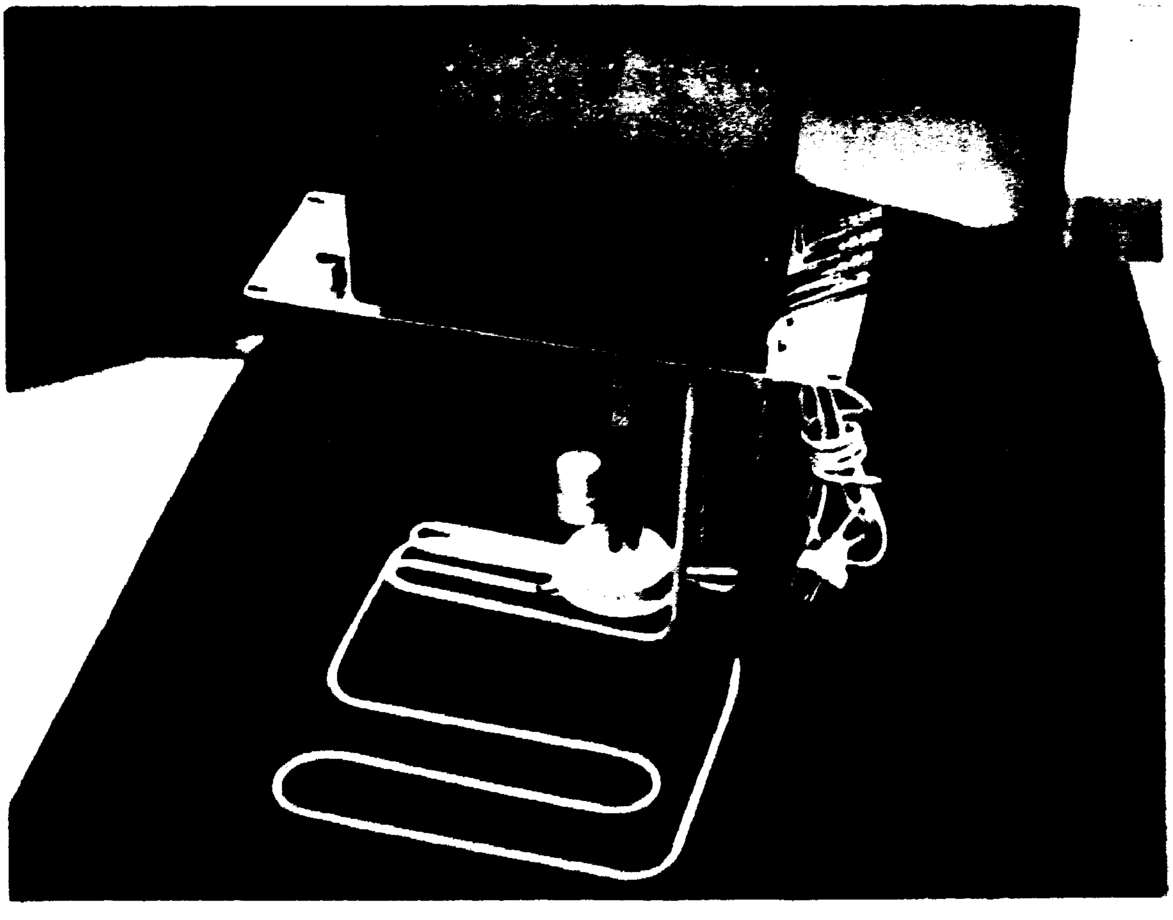

Fig.6. Hetotherm temperature control system.

5) As the etching bath should not be agitated (see 2 above) the temperature control unit cannot be located in the etching tank itself. Therefore, the etching tank was located inside a water bath, accommodating the temperature control system. As the etching bath is used also at temperatures above room temperature, the outside water bath (made of stainless steel) has a special heat insulation layer to avoid heat losses (see fig. 7.)

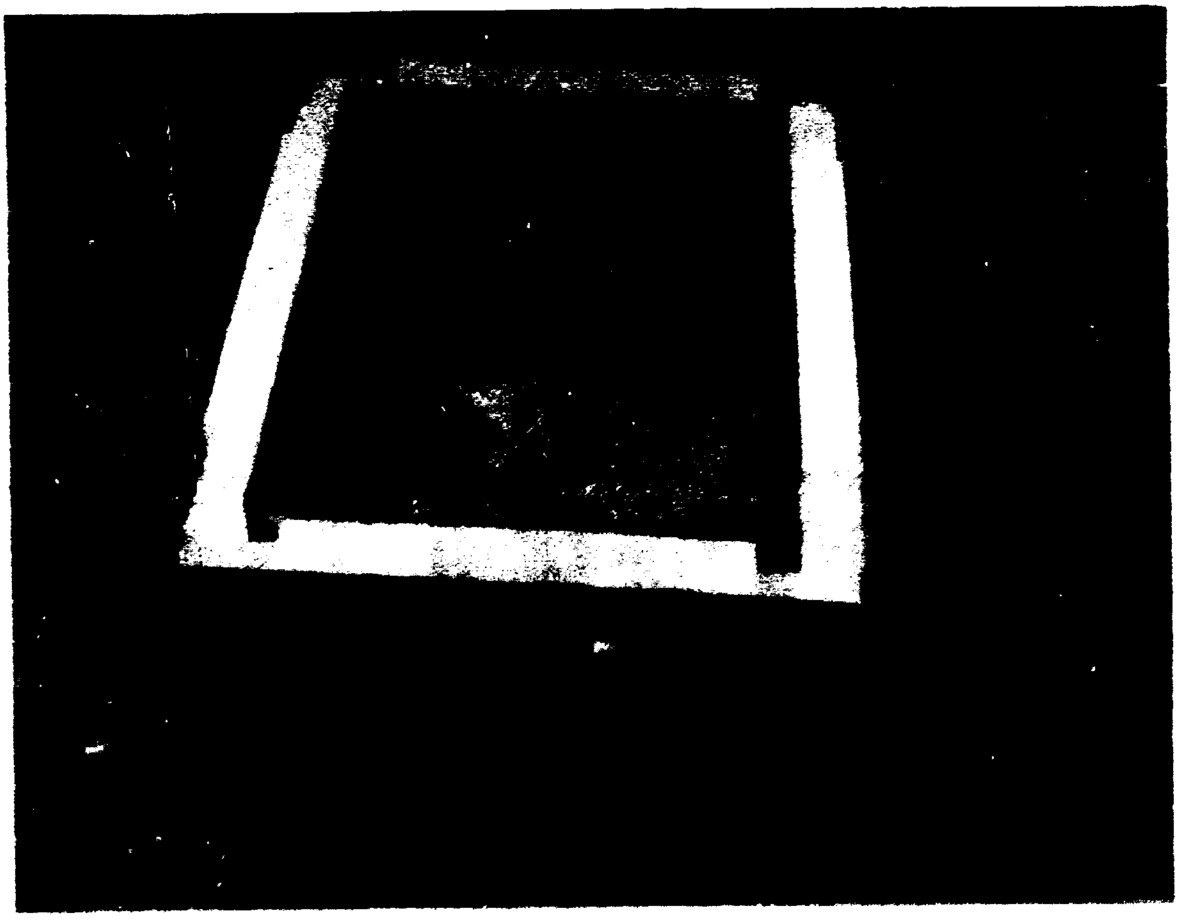

Fig. 7. Heat insulation of the water bath

The etching tank, together with the temperature control unit, hangs on a stainless steel lid inside the water bath. It can be lifted easily, as shown in fig. 8.

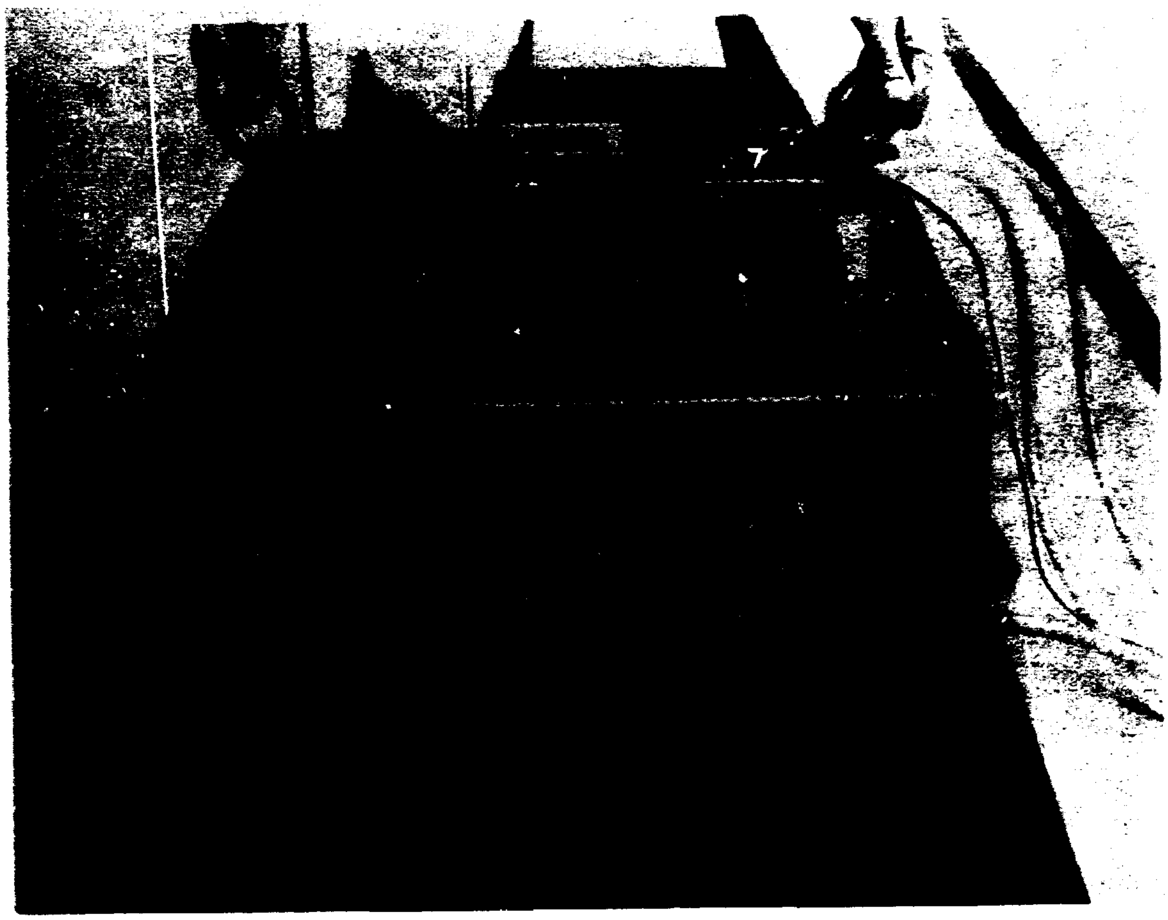

Fig. 8. Lifting of the etching tank from the water bath

The temperature of the water bath can be measured by means of a glass laboratory thermometer inserted through the lid of the water bath.

6. TEMPERATURE CONTROL

The Hetotherm temperature control unit (see Figs. 5 and 6) permits a very accurate temperature control (± 0.05°C). There are six ranges within which the temperature can be kept constant:

T1 - 25°C - 35°C
T2 - 20°C - 50°C
T3 - 35°C - 65°C
T4 - 50°C - 80°C
T5 - 65°C - 95°C
T6 - 80°C -110°C

These temperature ranges are marked on the front panel of the temperature control unit (see Fig. 9.)

The switch on the front panel chooses one of the ranges. Next the power swithch on the panel is put on and a neon lamp lights together with another lamp located besides it. This signifies that the power is on and that the heater too, is on.

Six trimmers are located on the side of the front panel, corresponding to the six temperature ranges. Through the holes located at the left side of the control unit (see Fig. 5) the desired temperature can be set by inserting a screw-driver and turning the inside potentiometer. When the desired temperature in the etching tank is reached it is signified by the right neon lamp on the front panel going off and on, which indicates also that the heater is switched on and off to keep this temperature constant.

To be able to hold a temperature lower than room temperature constant it is necessary to connect cooling water to the temperature control unit. This is done by means of two hoses connected to the tank of the temperature control unit (see Fig. 10.)

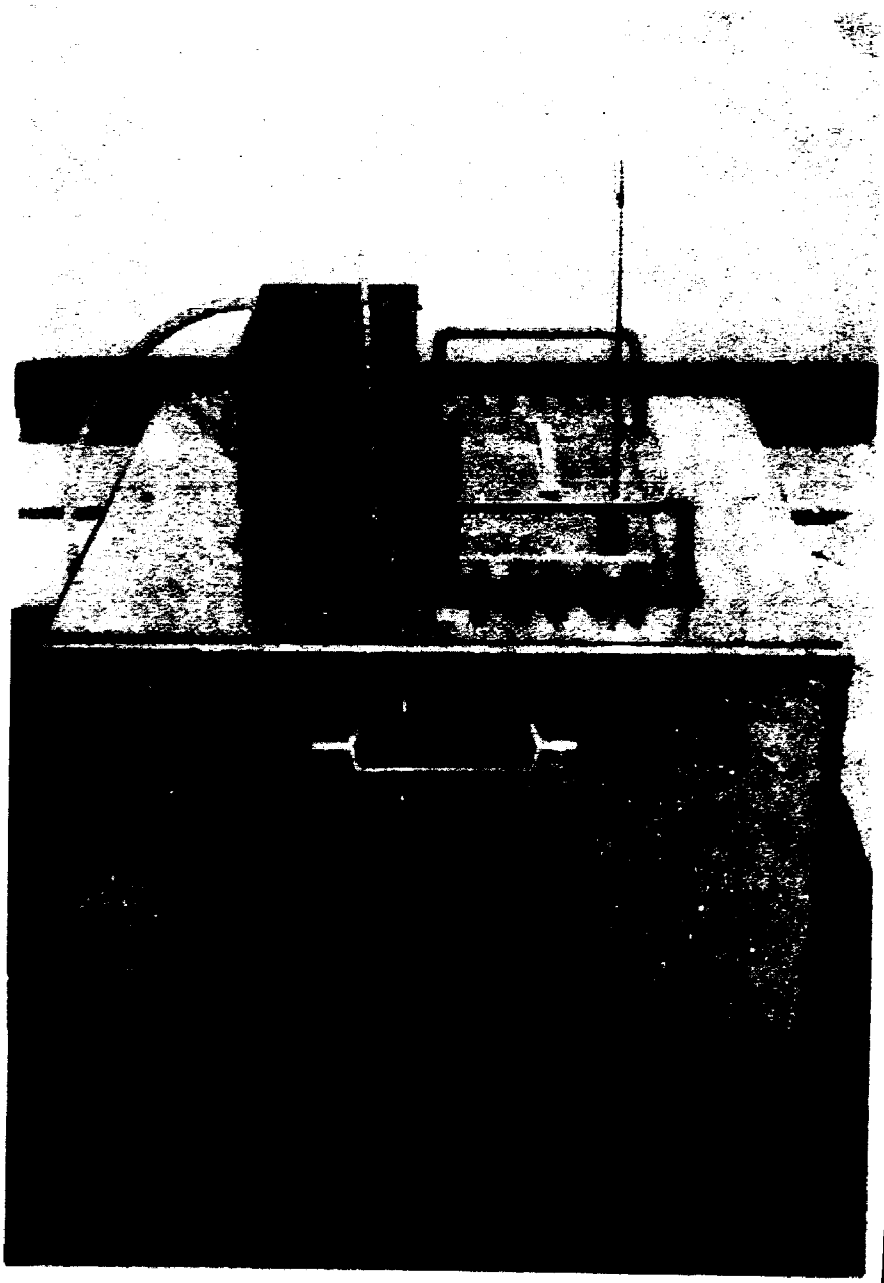

Fig. 9. Front view of the etching equipment

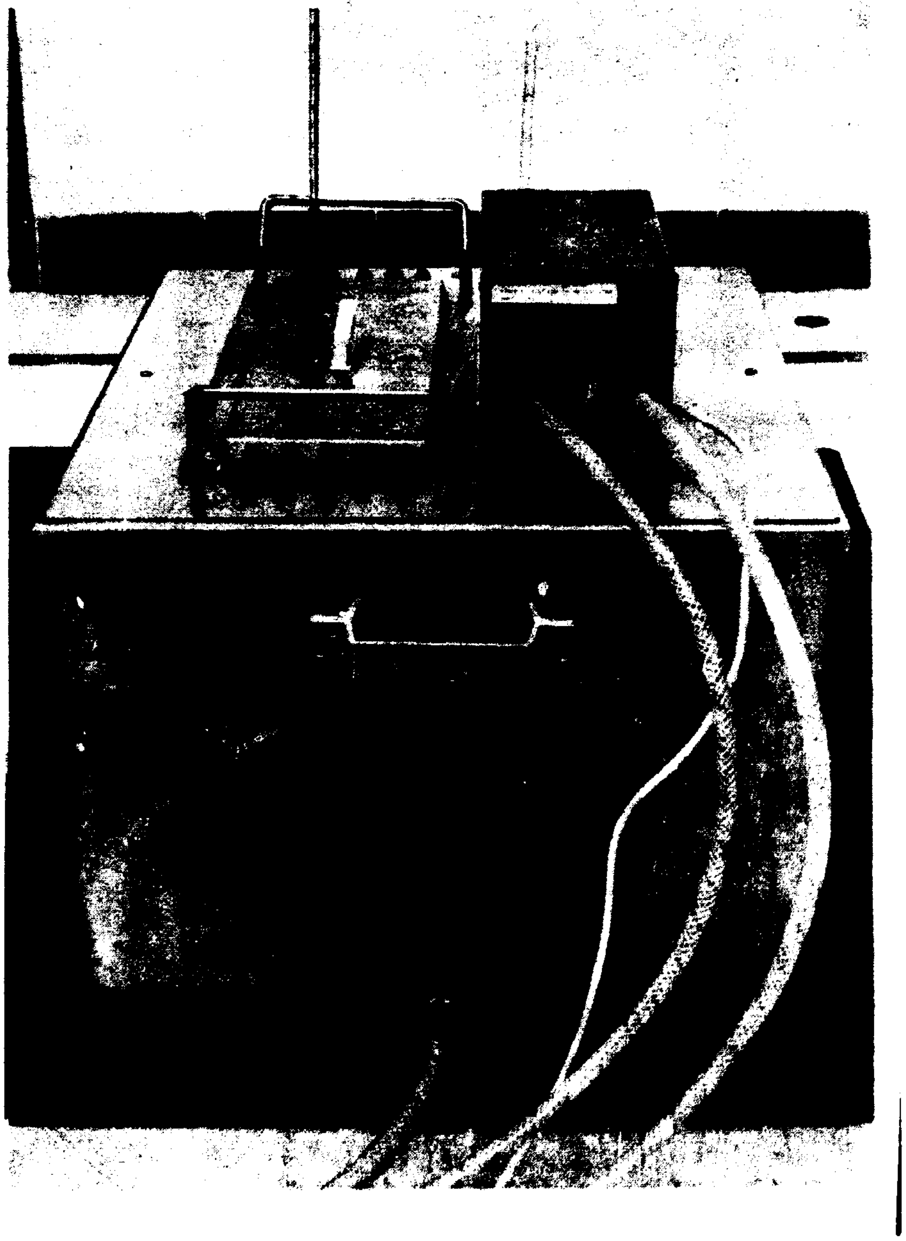

Fig. 10. Back view of the etching equipment

Through those hoses cooling water is connected to a inner cooling coil (seen in figs. 5 and 6.)

A water circulating pump of the temperature control unit (seen in figs. 5 and 6) assures the mixing of water inside the external water tank thus equalizing the temperature inside it.

A float (seen in figs. 5 and 6 at the left side of the water pump) prevents the heater to be switched on when there is no water in the external water bath.

7. DIMENSIONS AND CAPACITIES

The etching tank has the following dimensions:

- width: 140 mm,
- depth: 210 mm,
- length: 390 mm.

This tank can accommodate about 10 l of the etching solution, as it is not necessary to fill it to the brim.

The water bath has the following internal dimensions:

- width: 400 mm,
- depth: 305 mm,
- length: 485 mm.

Its external dimensions are:

- width: 510 mm,
- height: 390 mm,
- length: 595 mm.

It accommodates about 60 l of water. This water can be filled to the water tank through an opening in the lid (seen in the front of the temperature control unit in Figs. 9) and emptied through a valve, connected to a hose (seen at the bottom of the water tank in Fig. 10).

The total height of the whole equipment is 510 mm (to the top of the temperature control unit).

The required cooling water flow is about 10 l/min.

Power requirement: 1200 W, 220 V.

8. PERFORMANCE

Although the Kodak pamphlets describing the nitrocellulose film recommend an etching temperature of 60 and 25°C the Kodak-Pathé Research Laboratories at Vincennes, France have adopted a temperature of 50°C, while etching the nitrocellulose film for 45 min in a 10% sodium hydroxide solution.

At Risø National Laboratory it was found that best results can be reached while etching nitrocellulose film at 20° C for 21 h in the 10% of NaOH.

Therefore, the Euratom Neutron Radiography Working Group test program has adopted those two etching procedures: 45 min at 50°C and 21 h at 20°C.

The etching equipment was consecutively tested for temperature control at those two levels. With ambient temperature of 22°C it took 1 h 40 min to stabilize the temperature of the etching bath at 20°C with a flow of about 10 l/min of the cooling water, which had a temperature of 15°C.

Thereafter the etching bath temperature was raise to a stabilized temperature of 50°C in 2 h 30 min, with the cooling water shut-off.

Finally the etching bath was cooled down to the stabilized temperature of 20°C in 2 h with a cooling water flow of 10 l/min.

The stability of the temperature control was checked with an electric thermometer; inserted into the etching bath. This thermometer was connected to a strip-chart recorder, which registered the temperature in the etching tank. Figure 10 shows the stability of temperature during 20 min after it has reached the desired level of 20°C (bottom) and 50,1°C (top).

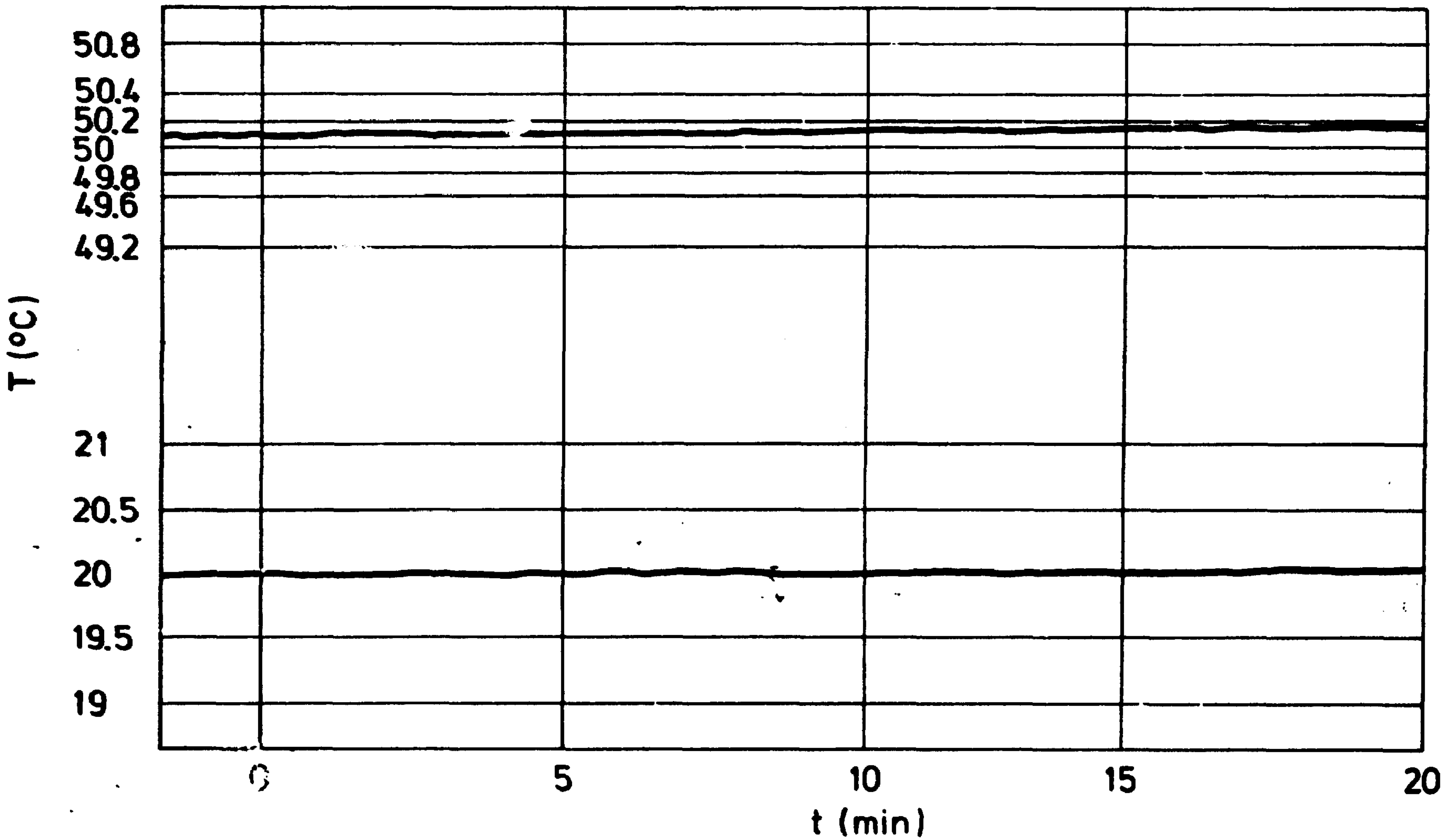

Fig. 10. Temperature stability at 20°C (bottom) and 50.1° (top)

During etching not only should the etching temperature be kept constant but the concentration of the etching solution as well.

As mentioned in 2 above the strength (concentration) of the etching bath affects the results obtained and it must therefore be prepared exactly and maintained at the correct strength. Preparing the etching bath with exactly the correct strength is comparatively easy, whereas to keep it at that strength at a higher temperature is not. Here one must allow for the effect of evaporation, which will affect the concentration of the etching bath.

This can be controlled by measuring the density of the etching bath with the help of a floating areometer. Figure 11 shows a set of such areometers from which one with the desired density range can be chosen.

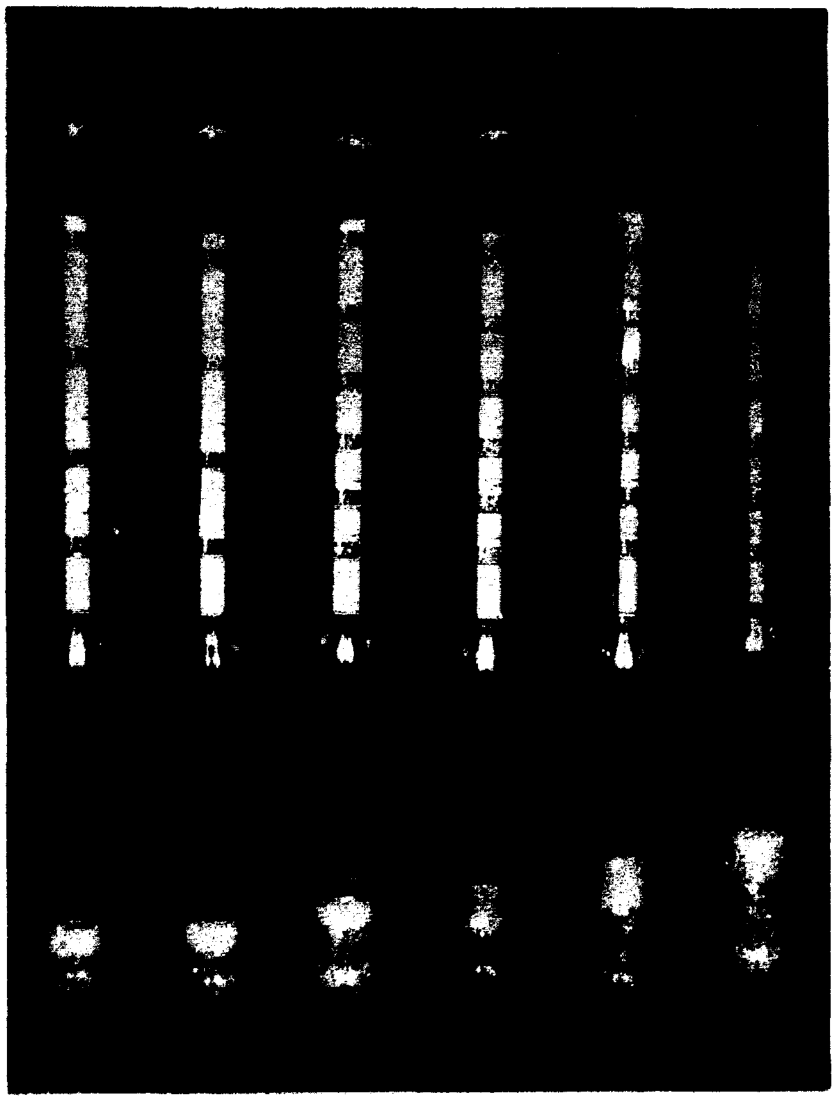

Fig. 11. A set of floating areometers

In Fig. 12 a curve is reproduced giving the relation of the density of a 10% solution of NaOH and temperature.

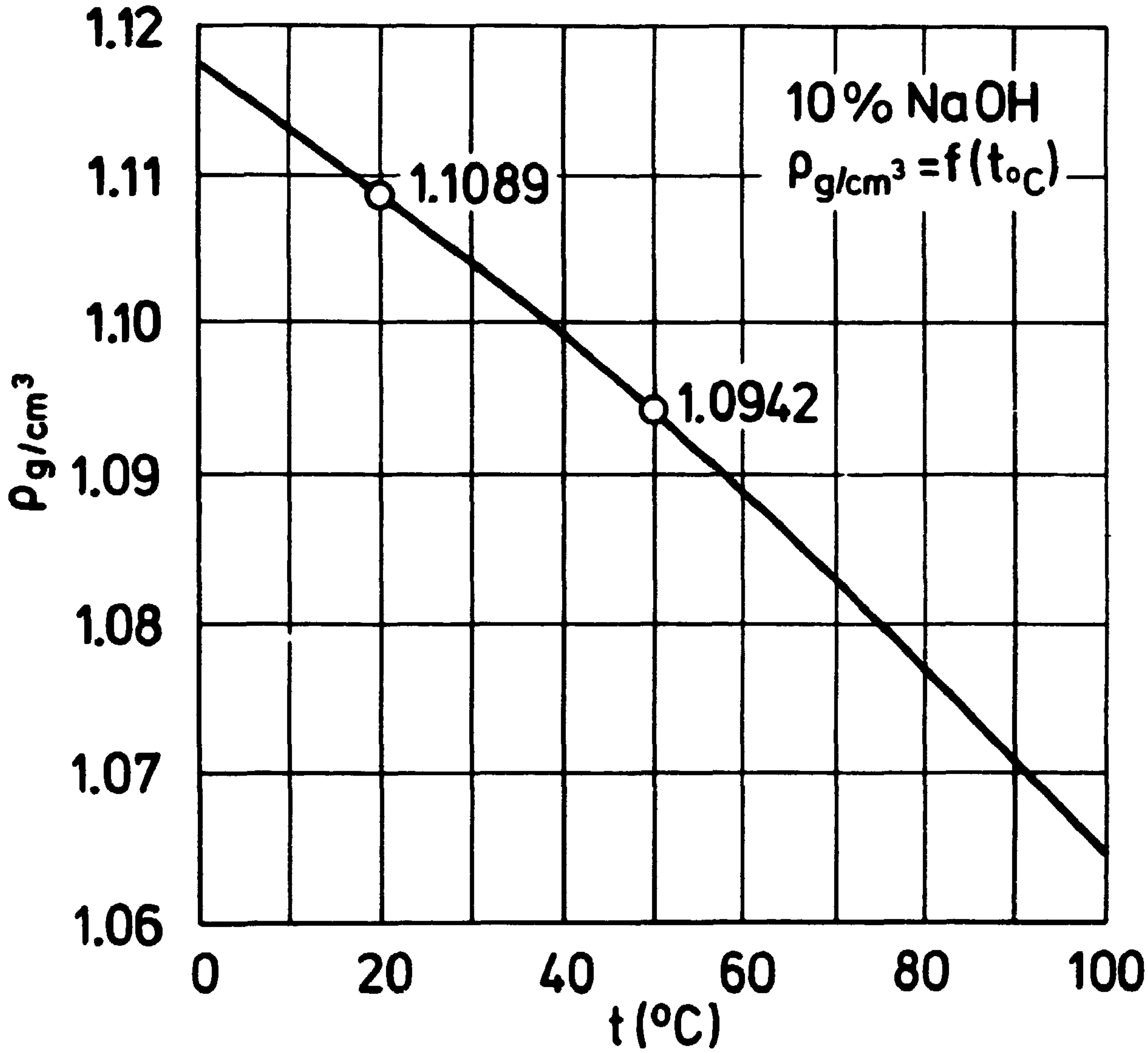

Fig. 12. Relation between density of a 10% NaOH and temperature

To control the concentration (density) of the etching solution it is sufficient to measure its temperature, and from Fig. 12 find the density corresponding to a 10% concentration of NaOH. If, due to evaporation, the measured density is higher than that shown in Fig. 12, it is sufficient to add so much water to the etching bath that its density will return to the correct one, shown in Fig. 12.

9. PRICE

As mentioned in 1 above, the equipment for etching nitrocellulose film is unavailable on the market and therefore Risø has designed and produced such special equipment. By doing so some typical parts were used. They consisted of the temperature control unit, manufactured by Heto Lab. Equipment A/S, Klintehøj Vænge 3, DK-3460 Birkerød. Heto has also supplied the stainless steel etching tank; the rest was produced at Risø.

The total cost of the whole equipment can be estimated as Dkr. 15,000.

Risø National Laboratory **Risø - M - 2396**

Risø - M - 2396

Title and author(s)

Special equipment for etching nitrocellulose film

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Date August 1983

Department or group

Metallurgy

Group's own registration number(s)

29 **pages** + 0 **tables** + 12 **illustrations**

Abstract

Nitrocellulose film and converter screens used for neutron radiography are described. Difficulties in visualization of radiographs on those films are mentioned. Because there is no equipment for etching nitrocellulose film available on the market Risø has designed and produced such equipment at an estimated cost of Dkr. 15,000. Design criteria for this equipment are given and its performance described.

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